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(54) **Process to refine raw waxy oils for alimentary use.**

(57) Process to refine raw waxy oils and particularly raw vegetable oils obtained from certain seeds, for example seeds of sunflower, corn, grapestone, and also husk oil resulting from the squeezing operation of olives: process which, different from those usually carried out to refine such types of oils for alimentary use, it comprises a sequence of phases suitably performed to eliminate directly both the free acidity and the wax contents, as well as the contents of impurities, further phases being then added for decolorizing and deodorizing purposes in order to obtain a final product for alimentary use which is limpid, bright, without disagreeable smell and taste. The refining process comprises first of all a set of operations by which the free fat acids and the waxy substances contained therein, as well as the different impurities are separated from the fed raw waxy oil before carrying out usual treatments for decolorizing and deodorizing purposes. The use of strong acid is not foreseen in this process.

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The object of the present invention is a refining process of raw waxy oils, and particularly raw vegetable oils obtained from some seeds, for example, those of sunflower, corn, grapestone, and also husk oil resulting from the squeezing operation of olives : process which, different from the refining process usually carried out to obtain such types of oils for alimentary use, comprises a sequence of phases suitably performed to eliminate directly both the free acidity and the contents of wax, as well as the contents of impurities, further operative phases being added for decolorizing and deodorizing purposes in order to obtain a final product for alimentary use, which is limpid, bright, without any disagreeable smell and taste.

In the last decania, the accentuated advertising and the possibility of obtaining oils of various seeds, the prime costs and, consequently, selling prices of which are commercially preferable, has greatly increased both the cultivation and extraction of raw oils, and the refining of these latter, by taking the aim of reducing as much as possible or, if possible to remove completely the free acidity as well as the waxy substances, to better the organoleptic features, make limpid the colour and eliminate disagreeable smell and taste.

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Referring to the raw waxy oils, the refining of which is the object of the present invention, processes are already provided in the prior art to eliminate, in a sequence of operative phases, the contents of free fat acids, waxy substances as well as various impurities, the presence of which may also remarkably lower the commercial value of the product, maybe because of disagreeable appearance, smell, taste...

In raw oil of sunflower, for example, a content of about 2% of free fat acids, and 0.2 to 0.3% of waxy substances is found. The impurities may include gummy substances and also metal particles. More diffused may be considered the alkaline refining process, indicated by the reference character A in the block diagram of the accompanying drawing, wherein a sequence of operative phases are provided, namely:

1. degumming
2. neutralization
3. decolorizing and filtering
4. dewaxing and filtering
5. deodorizing

In phase 1, the fed raw waxy oil is treated by means of water and strong acid, preferably phosphoric acid, at about 60°, in order to cause the precipitation of impurities (gummy substances and meta parts), so that they may be separated later through centrifugation.

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In phase 2 the neutralization of the product coming out from phase 1 is provided. The neutralization is carried out by means of caustic soda which reacts with the free fat acids, and a consequent making of soaps to be separated from the oil by centrifugation. A further washing and consecutive drying serve to complete the operation.

In phase 3 the semi-refined oil obtained through phase 2 is subjected to a treatment of decolorizing and filtering. This treatment is generally carried out with a suitable addition of activated carbon and the like, the scope of which is to absorb the colouring substances present in the oil during such refining phase, for example, chlorophyll, kerotene. The next filtration serves to separate these additives after the absorbing function of same has been carried out.

In phase 4, which concerns the dewaxing, the aim is taken to crystalize the waxy substances to better obtain the separation of same. The semi-refined oil obtained through phase 3 is to this end made cold during a suitable time (maturing) and then filtered with the aid of an adjuvant material (for example, fossil flour and the like).

According to this conventional process, the last phase 5 takes the aim of eliminating those volatile substances which are responsible for disagreeable smells and tastes. In this

phase the deodorizing treatment is carried out in a deodorizer with pre-heating and following heating at 230° to 250° and high vacuum, generally from 1 to 5 mm Hg.

For comparative purposes, suitable to evidence the operative and economic invention as explained hereafter, it is useful to point out that in accordance with above resumed conventional refining process of raw waxy oils indicated with the reference character A, the contents of waxy substances to attempt the greatest reduction or maybe the complete elimination of same is taken into consideration in phase 4 only.

Still in accordance with another process of alkaline refining of the prior art, the phase already indicated by the reference numeral 2 is substituted by a group of phases indicated by the reference character B in block diagram B. By such a process the aim of carrying out following operations is : to reduce, first of all, to about 10% the residual of soaps (coming out from phase 2a), without eliminating, maybe partially, the waxy substances : to remove the soaps nearly in their total contents and reduce to about 10% the contents of waxy substances (coming out from phase 4a): to eliminate totally the soap contents (coming out from phase 2a).

The semi-refined oil coming out from phase 2b, in accordance with this block diagram passes to phase 3a

concerning the deodorizing and filtering, the treatment being like that which was referred to in the cited process A, subjected only successively to a treatment 4b in order to complete the elimination of the waxy substances before the final phase 5, this latter being related to the deodorizing treatment of the product, and also like that already cited for a prior process A.

Another process, which is here indicated by the reference character C, is considered as a process of physical refining. By comparing this process C with process A as shown in block diagram, it is possible to note the lack of phase 2 concerning the treatment with caustic soda: it tends, nevertheless, to eliminate the free fat acids by suitably conforming the phase 5 previously cited exclusively for a deodorizing treatment : because of this conformation, such phase was further indicated by the reference numeral 5a.

These and other processes for refining raw waxy oils suggested and/or applied in accordance with the prior art by taking the aim of obtaining an oil for alimentary use, while tending to realize such a refining purpose with sufficient yield and satisfactory quality of the product, evidence some differences as regards the phase sequence of the treatments. These latter are specifically quoted in the block diagrams of the accompanying drawing, so that it is possible to note

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that phases substantially similar, are shown and indicated by respective reference numerals in accordance with those operative particularities which a person skilled in the art may consider as more suitable to reach the desired finality
5 and may then be selectioned in a better way on the ground of his own competence and experience.

Nevertheless, it is important to note that in the limited field comprising the most possible reduction, or better still, the total elimination of the free fat acids
10 and waxy substances present in the raw waxy oil, the prior art does not foresee the reaching of the desired results through an immediate sequence of treatments. To make the comparison easier, the examples of process according to the prior art as shown in block diagrams A,B,C of the accompanying
15 drawing, the reference numerals of the treatment phases, briefly hereabout, evidence that the treatment of dewaxing is foreseen as totally operating only after that concerning the deodorizing and filtering indicated by the reference numeral 3, in both processes A and C : the operation is near
20 total (about 90%) before phase 3 in process B, namely in the phase indicated by the reference numeral 4a, and then completely through the phase 4b, after the treatment of deodorizing is provided in such phase 3.

This course of the refining process has a more or less

negative influence upon the yield, installation and manufacturing costs, as well as upon the quality of the product, and makes further relatively more hard-working the refining process in its whole.

5 The invention as claimed, has the main scope of simplifying the refining process of raw oleaginous oils and is intended to remedy possible inconveniences which could obstruct the obtaining of alimentary oils having the desired good quality. It solves the problem of how to create a refining
10 process actually effective and reliable to eliminate totally the contents of free fat acids, waxy substances and various impurities already before subjecting the so purified product to final conventional treatments of deodorizing and decolorizing.

15 The advantages offered by present invention will still be more evidenced to the skilled in the art through the proposed sequence of phases which are explained hereafter in relation to the new refining process of raw waxy oils, as well as through the comparison with the process examples
20 concerning the prior art, by taking the aim of evidencing also the operative differences by looking through the black diagrams shown in the accompanying drawing.

In the drawing the reference numerals not having any

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added character are to be referred to conventional treatments in accordance with the premises also as regards the prior art, while through the added characters, differences relating to respective conventional treatments for a desired process are pointed out. Single reference characters are quoted in the block diagram concerning the process which is the object of the present invention, indicated in its whole by the reference character D, as briefly referred to above.

The refining process according to the present invention comprises, first of all, a set of operations by means of which the free fat acids and the waxy substances contained therein, as well as the various impurities are separated from the fed raw waxy oil, before carrying out next conventional treatments for decolorizing and deodorizing purposes. The operation may be resumed as follows :

- a; cooling treatment of the raw oil at the temperature of 0°C :
- b; mixing operation, in a conventional manner, with caustic soda to give rise to a reaction with the free fat acids and a consequent forming of soaps :
- c; a long maturing time i.e. a time which is necessary to form crystals of all the waxy substances contained in the oil being treated, and agglomerate these crystals both with the soaps and with the impurities contained

in so treated oil, in order to favour a total separation of same :

d. separation by centrifugal way, of the so formed agglomerat from the oil, so that this latter may

5 further be dried and then subjected to the next phase of:

e. decolorizing and

f. deodorizing

through conventional treatments already indicated by reference numerals 3 and 5, respectively.

10 Practical experiments of the applicant have evidenced that the phase B. of the refining process in accordance with the invention is to be carried out with caustic soda, the density of which is preferably about 10% higher than the stoichiometric calculus, by taking into account that the
15 soda density is to be selected on the grounds of the acidity of the oil to be treated, with a concentration increment based on the increment of acidity. Thus, for example, a soda concentration of 18° Bé may rightly answer to a content of free fat acids of about 1.5%, while a soda concentration
20 of 20° Bé may be in keeping with a content of about 3%.

In phase C the maturing time is extended to about more than 16 hours to reach those conditions which are suitable for forming crystals of the wax totality and obtaining the agglomeration as indicated above.

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This new refining process in accordance with the invention has also been shown by a block diagram indicated by the reference character D in the accompanying drawing. By comparing this latter with the block diagram of the process indicated by the reference character A it is possible to note that the phases of the process sequentially quoted from a to d replace those indicated by A., 2., 4. in the process of prior art and include the dewaxing treatment before the final treatments and particularly before the decolorizing treatment indicated by the reference numeral 3. This important difference does not relate to the refining process A only, but also to the processes B and C of the prior art.

It will easily be understood by a person skilled in the art, when he is looking through the block diagrams and considering carefully the novel features of the refining process in accordance with this invention, as described hereabout, that a substantial difference arises in respect to refining processes suggested and/or applied in accordance with the prior art. According to the process of the applicant, indicated by the reference character D, a particular importance is to be given to the selection of consecutive phases b and c, not only to realize the neutralization of the free acidity by itself, but mostly to succeed in

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obtaining, through a single agglomerate comprising the crystals of all the waxes in their making during these phases, the soaps resulting from the reaction of the caustic soda in its suitable concentration in respect to the acidity
5 of the oil being treated, as well as the various impurities whose separation takes place during such phases.

Furthermore, from the description of the operative phases a to f and particularly from that referred to the phase b, it gives a result that only the use of caustic
10 soda is provided, no strong acid being on the contrary employed. The resulting advantages should very clearly be understood by the skilled in the art. Particular mention is to be made for that which concerns the obtainable agglomerate as pointed out above, the separation of which
15 is performed by centrifugation, so that a remarkable simplification of the process is consequently possible.

As premised, the advantages concern both the installation and manufacturing costs, and the easier obtaining of refined oil having a better quality.

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CLAIMS

1. A refining process of raw waxy oils for alimentary use comprising treatments of : degumming ; neutralization of the free fat acids, dewaxing of the waxy substances ; elimination of the various impurities,
5 decolorizing and deodorizing ; characterized in that it comprises a sequence of operative phases on the oil which is fed for being refined in order to realize the total elimination of the free fat acids and waxy substances, as well as the separation of
10 the various impurities, all contained in the oil being treated before subjecting the obtained oil to the next phase of decolorizing and deodorizing, such sequences comprising :
 - a. cooling treatment of the raw waxy oil at the
15 temperature of 0°C :
 - b. mixing, in a conventional manner of the so cooled oil with caustic soda to provide the reaction with the free fat acids and the consequent forming of soaps :
 - 20 c. a long maturing time of the semi-refined oil as considered necessary and sufficient to favour both the forming of crystals from the waxy

substances totally contained in the oil being
treated, and agglomeration of such crystals, either
with the soaps made in said phase (b), and with the
various impurities, as well as those contained in
5 said oil ;

d. separation, by centrifugal way, of such agglomerated
whole from the oil to be further treated through the
following final phases of the process carried out
in conventional manner, namely :

10 e. decolorizing

f. deodorizing

2. Refining process as claimed in Claim 1, characterized
in that : the density of the caustic soda to be mixed
in accordance with said phase (b) is preferably
15 about 10% higher than the stoichiometric calculus;
the density of said caustic soda is to be selected
on the ground of the acidity of the oil being treated,
the increase of concentration being based on the
increase of acidity.

20 3. Process as claimed in Claim 2 characterized in that,
for example, a caustic soda concentration of 18° Bé
may rightly answer to a content of free fat acids of
about 1.5%, while a caustic soda concentration of

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20° Bé may rightly answer to a content of about 3%.

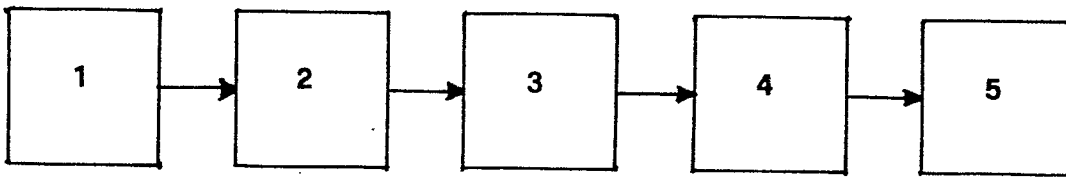
4. Process as claimed in Claim 1 characterized in that said long maturing time is extended up to about more than 16 hours.

5 5. Process as claimed in Claim 1, characterized in that in said degumming phase (a), neutralization phase (b) and dewaxing phase (c) is provided only the use of caustic soda, the employment of any strong acid being excluded.

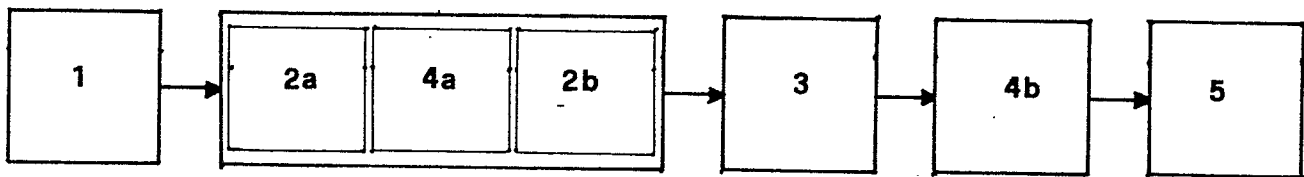
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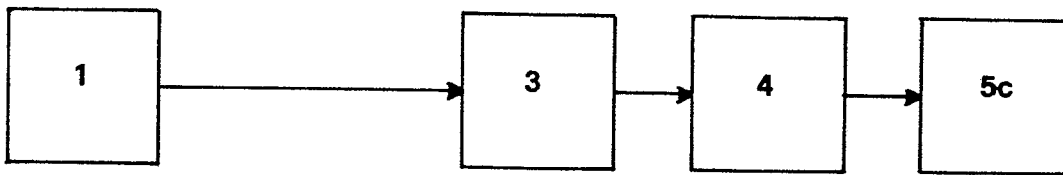
A



B



C



D

